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FIRST QUARTERLY PROGRESS REPORT

"Fabrication and Properties of Tungsten and Tungsten Alloy Single Crystals"

by the Staff of

Linde Company

and

Haynes Stellite Co.

Divisions of Union Carbide Corporation

For period June 29 to September 29, 1961

Contract: NOw 61-0671-C

For the Materials Branch,
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PREFACE

This work was done under the direction of Milton Stern, with major contributions from the following. At the Linde Company, R. W. Diesner conducted metallurgical observations, special heat-treatments, and recrystallization measurements; L. G. Tensmeyer and R. G. Rudness prepared the various special composition crystals. At the Haynes Stellite Co., J. W. Chasteen supervised fabrication and sample preparation for mechanical property measurements.

Early work described in the introduction was obtained in a cooperative program between the Linde Company and Union Carbide Metals Co. These data were obtained by E. L. Harmon, J. L. Wilson, and R. W. Fountain.

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SUMMARY

This is the first quarterly progress report of Contract NOw 61-0671-C and covers the period June 29 to September 29, 1961. The objective is to evaluate the fabricability and properties of tungsten and tungsten alloy single crystals. Approximately 60 crystals have been prepared with diameters from 1/2 to 1 inch and lengths ranging from 6 to 18 inches. Included in the program are several dilute alloys. The majority of crystals has been successfully swaged, forged or rolled at temperatures far below those required to fabricate polycrystalline tungsten. The influence of composition and orientation is currently being examined. The following properties are being determined on the fabricated material: hot hardness, recrystallization temperature, mechanical properties at elevated temperatures, and ductile to brittle transition temperature. When all the information is accumulated, it is expected that a clearer picture of the influence of interstitial elements on the above properties will be available.

I. INTRODUCTION

Many present and future materials problems require performance under extreme conditions of temperature and environment. This has led to extensive work involving tungsten and tungsten base alloys since the high melting point of these materials promises attractive strength properties if other problems specific to the material can be effectively solved. It is worth noting that some of the properties of tungsten which make it attractive for future application are often the same properties which create difficulty in furthering the application of this material. Major problems today involve efficient consolidation into dense metallic form, fabrication into useful shapes, and creation of unusually strong materials while minimizing the ductile to brittle transition temperature.

Consolidation of tungsten is primarily achieved by four different techniques. pressing and sintering, slip casting, consumable arc melting, and arc torch spraying. While each process produces final materials and shapes with some utility, many factors must be significantly improved before a completely satisfactory general process is achieved.

Today's techniques provide either low density, poor composition control, very poor mechanical properties, or low ductility. Also, they uniformly produce material which is very difficult to fabricate without extremely high temperatures and high deformation rates.[1] Consumable arc-melted tungsten requires extrusion at high deformation rates to break up the large grain size of the cast structure.[2] Even after such treatment, swaging must be done at temperatures as high as 1450°C (2640°F). Direct swaging of arc-melted tungsten requires temperatures in excess of 1700°C (3090°F).[3] Tungsten produced by powder metallurgical methods is swaged at temperatures of about 1500°C (2730°F). Hammer forging is started at about 1800°C (3270°F) with sheet rolling becoming practical at 1300°C (2370°F) to 1400°C (2550°F) after some direct forging for initial breakdown.[1]

The extreme conditions required to process tungsten are multiplied many fold for tungsten base alloys with much greater strengths at high temperatures. The technical possibility of creating tungsten alloys with significantly improved strength properties over tungsten has been established. However, the utility of such materials will never become a reality unless processing conditions which are realistic and reasonable can be established for their fabrication.

A portion of this program describes an approach with a high probability of success for achieving conditions which will permit ready fabrication of very high strength tungsten base alloys. The approach utilizes the improved fabricability of single crystals.

Samples of very small diameter tungsten single crystals grown by the strain anneal method have been known for some time[4] although property information has been limited and subject to some doubt because of incomplete characterization of the material. While extensive physical and mechanical property data for polycrystalline tungsten are reported[5], recent data on single crystals have not been available. Some preliminary data obtained on a cooperative program within the Union Carbide Corporation between the Linde Company and the Union Carbide Metals Company show some rather interesting and surprising effects on fabricability of tungsten single crystal. Table I presents results of swaging tests on as-grown crystals of various orientation. The tests were done on material of approximately 1/2-inch diameter. The orientations indicated represent the crystallographic direction along the axis of the cylindrical sample.

Swaging at 1000°C (1830°F) presented no problem. At 700°C (1290°F) partial reduction occurred easily but rapid cooling in the massive swaging machine caused a sufficient temperature drop to result in fracture of the samples. Since it is possible that as-grown crystals contain stresses which could influence their fabricability,[6] a few swaging tests were conducted after a stress relief at 1700°C (3090°F).

The tests recorded in Table II illustrate a remarkable difference in the fabricability of single crystal and polycrystalline tungsten. Single crystal material can be worked at from 500° to 700°C (900° to 1360°F) lower than conventional material. This is perhaps not surprising when one considers all the evidence for impurity segregation and second phase precipitation at grain boundaries.

Considerable data are now available on the mechanical properties of tungsten produced by various techniques.[7-11] Table III summarizes some of the pertinent data. The comparison is not yet complete or exact, since it is quite certain that impurities and surface preparation play a prominant role in determining mechanical properties - particularly at the lower temperatures. It is clear, however, that high purity single crystals have ductile to brittle transition temperatures well below room temperature when the surface is properly prepared. Also single crystals have high temperature strength properties comparable to polycrystalline tungsten of similar purity.

Table IV shows mechanical property data[3] for a few polycrystalline tungsten alloys tested at 1650°C (~3000°F). These alloys are among the strongest reported today at very high temperatures. Only two metallic materials in wire form are in the same strength region as the W-0.6% Cb alloy; a tungsten-30% rhenium alloy,[14] and single crystal tungsten containing 2% ThO_2 as a dispersed phase.[5]

The alloys in Table IV are not the result of extensive effort to arrive at optimum composition. Rather, they represent initial cursory trials, and yet, this work produced alloys with rather remarkable properties. Thus, it is highly probable that in the tungsten base alloy area undiscovered compositions exist with even more remarkable high temperature strength properties. When one considers alloys with such strengths, along with the difficulty of fabricating simple unalloyed tungsten, the conclusion appears likely that materials of this type cannot be handled practically by today's fabrication techniques. If the marked advantage in fabricability of single crystals shown in Tables I and II can be translated to alloy systems, the possibility of realizing practical and basic information becomes significant.

This report describes progress on a program for investigating tungsten and tungsten base alloys through use of a practical process for growing relatively large and adequately perfect single crystals of refractory metals. The program offers unique promise of important and rapid advances in the metallurgy of tungsten. It is designed to evaluate the influence of orientation, heat-treatment and substructure on fabricability; recrystallization behavior, hot hardness and mechanical properties at high temperatures of worked crystal; and the influence of interstitial impurities on certain of the above phenomena.

II. Experimental Methods

a. Crystal Growth

Tungsten and tungsten alloy crystals were grown by an arc modification of the Verneuil technique. The basic Verneuil method is described by Buckley[15]. Approximately 60 crystals were prepared for this program. Their diameters ranged from 1/2 to 1 inch with lengths varying from 6 to 18 inches. Most unalloyed crystals were 12 inches long. However, the alloy crystals and the high purity or high perfection crystals were generally 6 to 8 inches long. Typical samples are illustrated in Figures 1 and 2.

b. Composition

The chemical composition of several of the unalloyed crystals is shown in Table V. The results shown for metallic components are based on emission spectroscopy and are accurate to only ±50%. High purity compositions have not yet been analyzed.

Crystals with the following additions were also prepared: Nb, Zr, Ti, Hf, Ir, Ta, TaC, ThO_2, and $K_2SiO_2 + Al_2O_3$. The Zr, Hf and Ir alloys proved to be polycrystalline, and in the case of Zr, a two-phase structure resulted with concentration of Zr as low as 0.3%. Analysis of these materials is not yet available.

c) Working Techniques

Fairly conventional fabrication techniques were used successfully to swage, forge or roll, as compared to the rather complex methods required for polycrystalline tungsten.

Working was done either one of two ways. The first method consisted of centerless grinding followed by pickling in a 60-40 HNO_3-HF bath. After this treatment the crystals were then swaged. The other method was to pickle in the 60-40 HNO_3-HF bath then forge to a flat (about 50% the original diameter), pickle again and then roll.

This surface preparation was used to minimize fracture and cleavage since it was found that the removal of a small amount of the surface layer greatly increases the ductility of tungsten.[16]

The working schedule used was to preheat the specimens in a tube or muffle filled with argon followed by one pass in the die, forge or mill followed by a reheating to 1200°C. Analytical results indicate very little contamination of the material during working. (See PK, Table V.) The swaging went by 1/16 inch increments above 1/2 inch in diameter and by 1/32 inch increments below 1/2 inch in diameter. Most of the material was kept intact after a diameter of 3/8 inch was reached as this was the desired size for tensile specimens. A few sections of crystals were swaged down to .200 inch. Forging took the crystals down to about 1/2 their original diameters. About 3 reheats for the smaller diameter crystals (1/2 inch) and about 7 reheats for the large diameter crystals (1 inch) were needed. Hot rolling was done by increasing the draft .030 inch after each pass until the thickness of the sheet was about .040 inch. At this thickness the rolls were almost jammed. The thinnest sheet produced to date was about .031 inch thick.

During the working operations, various specimens or samples were removed from the crystals. A sample was cut from each crystal after completing a swaging die. Hardness readings were taken on these samples and in some cases the recrystallization temperature was determined. Samples were also removed after forging and various times during the rolling process. For hot hardness readings, specimens were removed after forging or after a few rolling passes. This was because of the requirements on specimen size in the hot hardness testing apparatus.

With the exception of a few specimens described elsewhere, all the crystals were either swaged to .375 inch or rolled to .040 inch from their original 1/2-inch diameter. This material was then sand- or metal-blasted to remove the oxide coating, sectioned, and then pickled.

The first few crystals that were forged and swaged were treated with various types of surface preparation. Those that had no pickling or centerless grinding seemed to have a greater tendency to fracture or cleave.

Figures 3 to 7 illustrate the appearance of typical materials at various stages of swaging, rolling or forging.

d) Metallography

The structure of the as-grown single crystals can be seen in Figures 8-11. Normally, crystals exhibit a cross section as shown in Figure 8. The boundaries between the "pie slices" are termed lineage here, if the amount of misorientation is less than about 5 degrees.

The misorientation is determined by the crystallographic angle between the normals of the exposed surfaces as determined optically or by means of a back reflection Laue photo. This method is acceptable for misorientation >1 degree; however, for smaller amounts of misorientation a microfocus X-ray tube is required and is currently being used. These areas change slightly as one goes from one end of the boule to the other. This can be seen by Figures 11a and 11b which are cross sections of the opposite ends of the same boule. In this case, the amount of misorientation increases and there is some coarsening of the substructure. There is almost always some change in the lineage but the amount and direction of change is not always the same. The reason for this is not fully understood but it is felt to be directly connected with the growth process.

Samples for various tests were sectioned appropriately. For the determination of the ductile brittle transformation temperature, bend specimens were cut from the .040 inch sheet so that the distance between loading points times the constant cross sectional area = .02 inch^3. This was done so that approximately a constant strain rate could be obtained. For tensile specimens, 2-1/4 inch lengths were cut from the .375 inch swaged rod and machined to standard size specimens. For hot hardness specimens, circular (or 2 semi-circular) disks 1-1/2 inch in diameter by 1/16 inch to 1/4 inch thick were cut. For room temperature hardness readings or recrystallization temperature determinations, specimens 1/4 to 1/2 inch long were cut from the swaged rod or sections 1/4 inch^2 or larger were cut from the rolled sheet.

Normal metallographic procedure was followed for all micrographs shown. In the case of the myriad of recrystallization specimens, a few short cuts were taken. The specimens normally were not mounted and were heavily etched after being polished on abrasive paper. While this often left some scratches on the surface, one could easily follow the progress of recrystallization. This was checked later by going through

a detailed diamond polishing procedure. The crystals were normally electrolytically etched in a 2% NaOH solution; however, a few of the alloys seemed to etch quite well in a 1:1 solution of HF - HNO_3. A fully recrystallized sample is shown in Figure 12.

If fracture occurred during working, it was relatively simple to determine if failure was caused by cleavage or boundaries. The difference is illustrated in Figures 13a and 13b. If the material had a high degree of misorientation or a second phase, it tended to fracture along boundaries. If the material became too cool during working or if there were stress risers present, the material tended to cleave.

III. Results

a) Fabrication Behavior

Tables VI and VII describe the behavior of various crystals during either swaging or forging and rolling.

Most of the fabricating was done at approximately the same temperature (1200°C). At this temperature three items seemed to have the most pronounced effect upon fabricability: chemical composition, amount of misorientation across boundaries, and orientation.

Chemical composition seemed to have an important effect, especially in the case of alloys. For example, an addition of 1% Zr or Ir produced extremely brittle material which couldn't be forged. Nb in the presence of over 50 ppm C and Hf additions also produced material which was brittle. Since the chemical analyses are as yet incomplete, no firm correlations can be drawn at the present time.

The amount of misorientation also appears to be fairly critical. This is anticipated in view of the known impossibility of fabricating polycrystalline tungsten under these conditions.

The effect of orientation on fabrication is not yet clear. Crystals PH and PK had about the same amount of misorientation and approximately the same chemical composition (see Table II). However, PK [110] kept fracturing while PH [100] swaged extremely easily. The reason for this is not well understood and will be investigated more fully.

b) Mechanical Property Data

From the material swaged to .375 inch, sections were cut and machined into tensile specimens for testing at room temperature and 1000°C.

All of the results obtained to date are listed in Table VIII. A number of the specimens broke during machining. Since only a limited number of tests have been completed, no definite conclusions can be drawn. However, compared to polycrystalline material (X-1) and data in the literature, the single crystals look quite good.

Hot hardness specimens have also been prepared and measurements are in process, but no results have been obtained to date.

Hardness values have been obtained on swaged material at various degrees of reduction. The trend of work hardening is shown in Figure 14. Some specimens have also been obtained from the material which was forged and rolled. While hardness values are not as complete in this area as for the swaged material, the trend seems to be the same. Work hardening is rapid in the early stages of reduction, followed by a relative insensitivity to further work.

Ductile-brittle transformation temperatures were determined by means of a bend test. Essentially three point loading was used. A .040 inch sheet was bent 90 degrees around a 3-T (3 times the thickness) radius. The transition temperature was determined on a go no-go basis. A list of the transition temperatures determined to date are listed in Table IX. A sample data sheet for one of the specimens is shown in Table X. The crystals (except for DB and DC) were all forged to about .250 inch from about .500 inch and then rolled to .040 inch. Crystals DB and DC were forged to about .450 inch from about .950 inch and then rolled (DB) or cross rolled (DC) to .040 inch. The bending apparatus is adjusted to a load rate of 15 pounds/minute. The specimen is prepared and mounted so that the strain rate is also approximately constant. Temperatures reported are for as-worked sheet followed by pickling. Some additional tests will be run on heat-treated specimens.

c) Recrystallization Behavior

Recrystallization temperatures based on isochronal anneals have been determined for most of the worked materials. These temperatures are listed in Table XI. A sample data sheet is shown in Table XII. The criterion used for recrystallization was 100% of the microstructure composed of recrystallized grains. Hardness readings in Rockwell A were also taken as a check; however, in some cases the material became so brittle that it shattered under the impact of the test. For this reason, it was felt that room temperature hardness readings could not be accurately used.

Results for representative samples, alloyed and unalloyed, are shown graphically in Figures 15a, 15b, 16a and 16b. From these graphs one can see the potential inconsistencies of using hardness readings exclusively. The progress of recrystallization is shown in Figures 17 and

18 for both unalloyed tungsten and alloyed tungsten.

On a number of the photographs the presence of deformation bands are noted; Figure 7, for example. Since they don't always appear, one would expect orientation to play an important effect in the deformation process and the properties obtained after deformation. This is further illustrated by the difference in recrystallization temperatures listed in Table XI. For example, PHA and PKA were both swaged to about 60% R.A. and PHA [100] had a recrystallization temperature of 1775°C while PKA [110] had a recrystallization temperature of only 1650°C. Another example is the difference in the degree of preferred orientation shown in Figure 19. Crystal PI is the only one which exhibits a true preferred orientation while the others exhibit a more random texture. A more critical examination of the effect of orientation is warranted.

DISCUSSION AND CONCLUSIONS

Since the work is in an incomplete stage, no definite conclusions can yet be stated. However, definite trends seem to appear. As far as fabrication is concerned, there definitely is an effect of low angle boundaries, chemical composition and orientation. The presence of large grain boundaries makes fabrication at low temperatures virtually impossible.

The effects of chemical composition appear more subtle. Certain alloying additions, Zr, Hf, Ir, cause severe embrittlement probably due to the formation of a second phase. The effect of interstitials and traces of metallic impurities is not yet clear. If the interstitial content is too high, >50 ppm of C, for example, then Nb alloys are almost impossible to fabricate. With high purity unalloyed tungsten no correlation is yet available.

Crystallographic orientation is another factor which definitely seems related to deformation characteristics and physical properties after working. This too needs further study; however, it appears as if [100] crystals have a higher recrystallization temperature and a lower ductile-brittle transformation temperature. The reason for this is not clear but may be related to the fact that the amount of "easy glide" is increased so that essentially one has less work hardening.[17]

REFERENCES

(1) V.D. Barth, "The Fabrication of Tungsten," DMIC Report 115, OTS PB 151071, August 14, 1959

(2) F.A. Foyle, G.E. McDonald & N.T. Saunders, "Initial Investigation of Arc Melting and Extrusion of Tungsten," NASA, TND-269, March 1960

(3) Staff of Union Carbide Metals Co., "Investigation of the Properties of Tungsten and Its Alloys," WADD Technical Report 60-144, May 1960

(4) F.S. Goucher, "Studies of Tungsten Single Crystals," Philosophical Magazine, 48, 229 (1924); 50, 289 (1926)

(5) V.D. Barth, "Physical and Mechanical Properties of Tungsten and Tungsten-Base Alloys," DMIC Report 127, March 15, 1960, OTS PB 151084

(6) Private Communication, Dr. R.W. Fountain, Metals Research Laboratory, Union Carbide Metals Co.

(7) R.H. Atkinson, "Physical Metallurgy of Tungsten and Tungsten Base Alloys," WADD TR 60-37 AD 240981, May 1960

(8) V.D. Barth, "Review of Recent Developments in the Technology of Tungsten," DMIC Memo 108, OTS PB 171627, May 18, 1961

(9) H. Braun and K. Sedlitschek, "On the Influence of Small Additions of Metals and Nonmetals on the Sintering, Working and the Mechanical Properties of Tungsten," Journal of Less Common Metals, Vol. 2, No. 2-4, April/Aug. 1960, p. 277-291

(10) R.I. Jaffee, W.J. Harris, Jr., N.E. Promisel, "Development of Refractory Metal Sheet in the United States," Journal of the Less Common Metals, Vol. 2, No. 2-4, April/Aug. 1960, p. 95-103

(11) S.J. Noesen and J.R. Hughes, "Arc Melting and Fabrication of Tungsten," Trans. AIME Vol. 218, April 1, 1960, p. 256-61

(12) Unpublished Data, Union Carbide Metals Co.

(13) H.W. Schadler, "Deformation Behavior of Zone-Melted Tungsten Single Crystals," Trans AIME, 218(4), 649-55 (1960)

(14) C.H. Sims and R.I. Jaffee, "Properties of Refractory Alloys Containing Rhenium," Trans. ASM, 52, 929 (1960)

REFERENCES Cont'd.

(15) H.E. Buckley, "Crystal Growth," John Wiley and Sons, Inc., New York (1951) p. 98

(16) J.R. Stephens, "Effect of Surface Conditions on Ductile-to-Brittle Transition Temperature of Tungsten," NASA Technical Note D-676, February 1961

(17) A. Seeger, J. Diehl, S. Moder, H. Rebstock, "Work-Hardening and Work-Softening of Face-Centered Cubic Metal Crystals," Philosophical Magazine, Vol. 2, VIII #15, March 1957, p. 323

TABLE I

SWAGING TESTS WITH SINGLE CRYSTAL TUNGSTEN

WITHOUT PRIOR ANNEAL*

Orientation	Temperature	Observation
(110)	600°C (1110°F)	N.G.
(110)	700°C (1290°F)	15.7% partial reduction before breaking
(100)	700°C (1290°F)	38.5% partial reduction before breaking
(111)	700°C (1290°F)	35.1% partial reduction before breaking
(100)	1000°C (1830°F)	55% reduction, swaged easily
(111)	1000°C (1830°F)	37% reduction, swaged easily
(210)	1000°C (1830°F)	51% reduction, swaged easily

* Powder metallurgy W requires about 1500°C (2730°F) while arc-melted W requires about 1700°C (3090°F) to swage. The swaging temperature of arc-melted W after impact extrusion can be lowered to 1450°C (2640°F).

TABLE II

SWAGING TESTS ON SINGLE CRYSTAL TUNGSTEN

AFTER STRESS RELIEF AT 1700°C FOR ONE HOUR

Orientation	Temperature	Observation
(100)	900°C (1650°F)	Swaged easily
(100)	~700°C (1290°F)	Swaged easily

TABLE III

SOME PROPERTIES OF TUNGSTEN PREPARED BY VARIOUS METHODS

Property	*Linde Single Crystal[12]	Powder Met.	Zone-Refined Single Crystals	Arc Melted
U.T.S. at 150°C	118, 300	124, 800[5]		122, 200[5]
Y.S. at 150°C	105, 100	123, 800		119, 100
Red Area at 150°C	6.0%	1.2	>10%[13] 0-16%[7]	1.9
U.T.S. at 1200°C	35, 000	25, 000[7]		55, 000[12]
Y.S. at 1200°C	34, 000	12, 000		52, 000
Elong. at 1200°C	10%			13.0%
U.T.S. at 1650°C	10, 000	13, 000[12]		14, 000[12]
Y.S. at 1650°C	10, 000	7, 500		9, 800
Elong. at 1650°C	**	32%		42%

* Approximately 1/2 inch diameter arc Verneuil grown crystal. Tensile sample surface mechanically ground.

** Chisel-like deformation

TABLE 1V

MECHANICAL PROPERTY TESTS OF W ALLOYS--POLYCRYSTALLINE

1650°C (~3000°F) Strain Rate 0.02 min.$^{-1}$

	Yield 0.2% Offset psi	Ultimate Tensile Strength psi	Elongation %	Reduction Area %
Arc Melted W	9,800	14,000	41.8	98
Powder Metallurgy W	7,500	13,400	32.0	40.5
W + 0.005% Ti	13,700	17,100	36.0	98
W + 0.12% Zr	40,800	47,700	16.0	38.9
W + 0.57% Cb	50,000	60,600	20.2	81.7
W + 0.88% Cb	45,000	46,100	15.4	84.5

TABLE V

CHEMICAL ANALYSIS OF UNALLOYED TUNGSTEN SINGLE CRYSTALS

Crystal and Orientation Designation	Oxygen PPM	Carbon PPM	Fe %	Si %	Mo %	Ni %	Ca %	Cu %	Mg %	Al %	Cr %
Starting Powder	400	70	.05	.05		.05	.005	.0005	.005		.00
PA [111]	22	24	.005	.005	.05	.005					
PB [111]	20	30			.05	.005					
PC [111]	23	17	.005	.005							
PD [100]	2	11			.05	.005					.005
PE [111]	24	10			.05	.005					
PF [210]	6	8			.05	.005					
PG [111]	12	24	.005	.005							
PH [100]	10	41									
PI [100]	2	22	.005							.005	.005
PJ [110]	4	4	.005		.005						
PK [110]	14	16									
* PK [110]	8	9									
* PK [110]	26	6		.005							
PL [210]	14	18			.005						
PM [210]	13	22									
PN [112]	8	21	.005								
DA [111]	4	9			.05			.0005			
DD [111]	5	12	.005		.005						

* Analysis after swaging and repeated anneals in argon.

TABLE VI

THE BEHAVIOR OF TUNGSTEN AND TUNGSTEN ALLOY SINGLE CRYSTALS DURING SWAGING TO ABOUT 50% R.A.

Crystal	Axis	Alloy Addition	Swaged Well	Fractured Slightly at Ends Only	Fractured or Cleaved Completely	Remarks
						Composition of alloys is nominal since analysis is not yet available.
PAA	111	None			X	No surface preparation; large misorientation
PAB	111	None			X	No surface preparation; vacuum annealed at 1800°C for 1 hour, cleaved in half at 20% R.A.
PAC	111	None		X		No surface preparation; less misorientation than PAA
PBA	111	None	X			Centerless ground; some surface cracks appeared but were ground out
PBB	111	None	X			Centerless ground; pickled; vacuum annealed at 1800°C for 1 hour
PBC	111	None	X			Centerless ground; pickled, a small section was swaged to 89% R.A.
PCA	111	None	X			Pickled
PCB	111	None		X		Pickled; one end fractured due to misorientation
PCC	111	None		X		Pickled; vacuum annealed at 1800°C for 1 hour

- continued -

TABLE VI Cont'd.

THE BEHAVIOR OF TUNGSTEN AND TUNGSTEN ALLOY SINGLE CRYSTALS DURING SWAGING TO ABOUT 50% R.A.

Crystal	Axis	Alloy Addition	Swaged Well	Fractured Slightly at Ends Only	Fractured or Cleaved Completely	Remarks
PDB	100	None			X	All the rest of the crystals on the list have been centerless ground and pickled. This one cleaved along cracks started during grinding.
PEB	111	None	X			
PFB	210	None		X		Slight surface cracking one end large misorientation.
PH	100	None	X			Swaged easiest.
PK	110	None		X		Cracking believed to be partially caused by orientation.
PL	210	None		X		
PN	112	None	X			
DA	111	None			X	Believed to be mainly caused by misorientation; possibly permitted to get too cold (0.945 inch)
DD	111	None		X		Swaged to .375 inch from .903 inch.
RA	111	High Purity	X			
RC	111	High Purity	X			

-- continued --

TABLE VI Cont'd.

THE BEHAVIOR OF TUNGSTEN AND TUNGSTEN ALLOY SINGLE CRYSTALS DURING SWAGING TO ABOUT 50% R.A.

Crystal	Axis	Alloy Addition	Swaged Well	Fractured Slightly at Ends Only	Fractured or Cleaved Completely	Remarks
RE	111	High Perfection	X			
DE	111	1% Ta		X		Possibly due to misorientation or chemical composition
DH	111	1% Ti	X			
DI	111	1% Zr			X	This was not a single crystal.
DK	111	2% ThO_2	X			
DM	111	1% $K_2SiO_2 + Al_2O_3$	X			
DO	111	0.38% TaC		X		Possibly a combination of misorientation and chemical composition.
DW	111	0.3% Zr			X	This was not a single crystal.
DX	111	3% ThO_2	X			
RH	111	1% Hf			X	This was not a single crystal.
RK	111	1% Nb	X			Believed to have a very low interstitial content.
RO	111	1% Nb		X		Believed to be caused by interstitial content.
RP	111	1% Nb		X		Believed to be caused by interstitial content; vacuum annealed at 1800°C for 1 hour, no appreciable difference noted.

- continued -

TABLE VI Cont'd.

THE BEHAVIOR OF TUNGSTEN AND TUNGSTEN ALLOY SINGLE CRYSTALS DURING SWAGING TO ABOUT 50% R.A.

Crystal	Axis	Alloy Addition	Swaged Well	Fractured Slightly at Ends Only	Fractured or Cleaved Completely	Remarks
RR	111	1% Nb			X	Believed to be caused by high interstitial content.
RS	111	1% Nb			X	Believed to be caused by high interstitial content.
X1	Polycrystalline	None		X		Commercial purity swaged tungsten rod added to program for comparison.

TABLE VII

THE BEHAVIOR OF VARIOUS TUNGSTEN AND TUNGSTEN ALLOY CRYSTALS DURING FORGING TO ABOUT 1/2 ORIGINAL DIAMETER FOLLOWED BY ROLLING TO .040 INCH

Crystal	Axis	Alloying Agent	Forged Well	Broke During Forging	Rolled Well	Some Tearing During Rolling	Remarks
PG	111	None	X	X	X		One section which wasn't pickled broke during forgin
PI	100	None	X		X		
PJ	110	None	X			X	Possibly due to orientation
PM	210	None	X		X		
PDA	100	None	X		X		
PEA	111	None	X		X		
PFA	210	None	X		X		
DB	111	None	X		X		
DC	111	None	X			X	Cross rolled
DF	111	1% Ta	X		X		
DG	111	1% Ti	X		X		
DJ	111	1% Zr		X			Polycrystalline
DL	111	2% ThO_2	X		X		
DN	111	1% K_2SiO_2 + Al_2O_3	X		X		
DP	111	0.38% TaC	X			X	Possibly due to chemical composition
RB	111	High Purity	X		X		
RD	111	High Purity	X		X		
RF	111	HighPerfection	X		X		
RG	111	1% Hf		X			Polycrystalline
RI	111	1% Ir		X			Polycrystalline
RL	111	1% Nb	X		X		
RU	111	1% Nb	X			X	Probably due to high interstitial content
RV	111	1% Nb	X			X	Probably due to high interstitial content
RT	111	1% Nb	X		X		
X2	110	None	X		X		Electron beam zone refined single crystal

TABLE VIII

TENSILE PROPERTIES AT 1000°C OF TUNGSTEN AND TUNGSTEN ALLOYS SWAGED FROM SINGLE CRYSTALS

BAR STOCK

Identification	Alloying Agent	Growth Direction	Per Cent Work	Yield Strength (psi) X 10^{-3}	Ultimate Strength (psi) X 10^{-3}	Per Cent Elongation in 1-in.
X-1	None	Polycrystalline	46	55.7	61.0	92.0
PB	None	(111)	54	66.7	66.9	87.0
PN	None	(112)	56	57.8	57.9	84.0
PK	None	(110)	58	56.6	56.7	85.0
PL	None	(210)	53	48.8	49.0	82.0
PH	None	(100)	54	45.3	45.3	83.6
RA	High Purity	(111)	58	58.0	58.0	88.0
DK	ThO_2	(111)	41	59.7	59.7	72.0
DE	Ta	(111)	42	75.1	75.1	90.0
DM	K_2SiO_2 + Al_2O_3	(111)	46	38.6	38.6	23.0

TABLE IX

DUCTILE-BRITTLE TRANSFORMATION TEMPERATURE

Specimen	Axis	Alloying Agent	Transition Temperature °F	Transition Temperature °C	Remarks
PGA	111	None	525	275	Not pickled prior to test
PGB	111	None	575	300	Not pickled prior to test
PGC	111	None	425	230	Pickled prior to test
PJA	110	None	425	230	
PJB	110	None	425	230	
PM	210	None	475	250	
PI	100	None	400	215	
PIX	100	None	525	275	Cross rolled; transverse specimen
DB	111	None	375	200	
DC	111	None	575	300	Cross rolled; transverse specimen
DF	111	1% Ta	525	275	
DG	111	1% Ti	475	250	
DL	111	1% ThO_2	575	300	
DN	111	K_2SiO_2 + Al_2O_3	375	200	
DP	111	0.38% Ta	550	285	
RB	111	High Purity	500	260	
RD	111	High Purity	500	260	
RF	111	High Perfection	575	300	
RV	111	1% Nb	550	285	
RT	111	1% Nb	550	285	
RL	111	1% Nb	500	260	

TABLE X

DATA SHEET FOR DETERMINING TRANSITION TEMPERATURE UNALLOYED CRYSTAL PI [100]

Temperature °F	Test Results *	
250	O, X, X, X	
300	O, O, O, +, X	
350	X, X, X	
400	O, X	Transition Temperature
450	O, O	
500	O, O	

* X - Specimen fractured
O - Specimen bent with no cracking
+ - Specimen bent with some cracking

The reason for some of the crystals bending at 300°F is not yet known. Chemical analyses were obtained on a specimen which bent at 300°F and one which fractured at 350°F. No difference was observed. This will be pursued further.

TABLE XI

RECRYSTALLIZATION TEMPERATURES OF WORKED SINGLE CRYSTALS

Crystal	Axis	Alloying Agent	Type of Working	Recrystallization Temperature °C
PAC	111	None	62% R.A. Swaging	1700 ± 35°
PAB	111	None	20% R.A. Swaging	1900
PBA	111	None	56% R.A. Swaging	1750
PBB	111	None	58% R.A. Swaging	1700
PBC	111	None	56% R.A. Swaging	1700
PB	111	None	85-90% R.A. Swaging	1625
PCA	111	None	60% R.A. Swaging	1750
PEB	111	None	62% R.A. Swaging	1700
PFB	210	None	63% R.A. Swaging	1700
PKA	110	None	60% R.A. Swaging	1650
PKB	110	None	10% R.A. Swaging	2025
PHA	100	None	60% R.A. Swaging	1775
PL	210	None	61% R.A. Swaging	1675
PNB	112	None	62% R.A. Swaging	1625
RA	111	High Purity	56% R.A. Swaging	1575
RC	111	High Purity	50% R.A. Swaging	1800
RE	111	High Perfection	60% R.A. Swaging	1675
DE	111	1% Ta	55% R.A. Swaging	1825
X1	Polycrystalline	None	50% R.A. Swaging	1775-1800
DH	111	1% Ti	52% R.A. Swaging	1775
DK	111	ThO_2	52% R.A. Swaging	1900
DM	111	K_2SiO_2 + Al_2O_3	55% R.A. Swaging	1725
DO	111	0.38% TaC	50% R.A. Swaging	1900
RO	111	1% Nb	47% R.A. Swaging	2125
RP	111	1% Nb	49% R.A. Swaging	2075
RK	111	1% Nb	51% R.A. Swaging	1900
RH	111	1% Hf	20% R.A. Swaging	1525
PGC	111	None	As forged	1650 *

* This value seems low but it was rechecked by an additional anneal. Perhaps during hot-rolling some stress is relieved.

- continued -

TABLE XI Cont'd.

RECRYSTALLIZATION TEMPERATURES
OF WORKED SINGLE CRYSTALS

Crystal	Axis	Alloying Agent	Type of Working	Recrystallization Temperature °C	
PGA	111	None	Rolled to .042 in.	1750	
PGB	111	None	Rolled to .041 in.	1750	
PJA	110	None	Rolled to .040 in.	1675	
PJB	110	None	Rolled to .041 in.	1625	
PIA	100	None	Rolled to .056 in.	1700	
PIx	100	None	Cross rolled to .048 in.	1725	
PMA-1	210	None	Rolled to .120 in.	1775	
PMA-2	210	None	Rolled to .046 in.	1700	
PMB-1	210	None	Rolled to .032 in.	1675	
DBA	111	None	Rolled to .046 in. from .462 in.	1725	
DBB	111	None	Rolled to .195 in. from .462 in.	1725	
DC	111	None	Cross rolled to .045 in. from .467 in.	1750	
RB	111	High Purity	Rolled to .042 in.	1725	
RD	111	High Purity	Rolled to .043 in.	1875	
RF	111	High Perfection	Rolled to .042 in.	1725	
DF	111	1% Ta	Rolled to .042 in.	1850	
DG	111	1% Ti	Rolled to .042 in.	1950	
DL	111	2% ThO_2	Rolled to .042 in.	1725	
DN	111	K_2SiO_2 + Al_2O_3	Rolled to .042 in.	1750	
DP	111	0.38% TaC	Rolled to .043 in.	1825	
DX	111	3% ThO_2	Rolled to .044 in.	>1850	Not complete
RL	111	1% Nb	Rolled to .044 in.	1850	Not complete
RU-1	111	1% Nb	Rolled to .045 in.	2000	
RU-2	111	1% Nb	Rolled to .063 in.	2000	
RV	111	1% Nb	Rolled to .044 in.	1925	
RT	111	1% Nb	Rolled to .061 in.	1875	

TABLE XII

SAMPLE DATA SHEET USED FOR RECRYSTALLIZATION TEMPERATURES

Temperature °C	PMA-1 [210] Unalloyed Rolled to .120 inch		DN [111] $K_2SiO_2 + Al_2O_3$ Rolled to .042 inch		RU-1 [111] 1% Nb Rolled to .045 inch	
	Hardness R_A	Micro*	Hardness R_A	Micro*	Hardness R_A	Micro*
Room Temperature	68.6	0	71.6	0	71.7	0
1250	65.1	25	66.4	5		
1350	61.7	40	63.1	60	69.5	0
1500	60.9	60	63.4	60	66.3	20
1620	61.2	75	60.9	80	63.6	30
1700	60.9	85	61.7	95		
1775	61.2	100	60.0	100	64.1	80
1850					63.7	85
1975					63.0	98-100
2100					62.7	100
Recrystallization Temperature	1775		1750		2000	

* Micro=percent recrystallized by microscopic observation

Fig. 1 - Single crystal of tungsten - 3/4 inch in diameter and 12 inches long. Normal size.

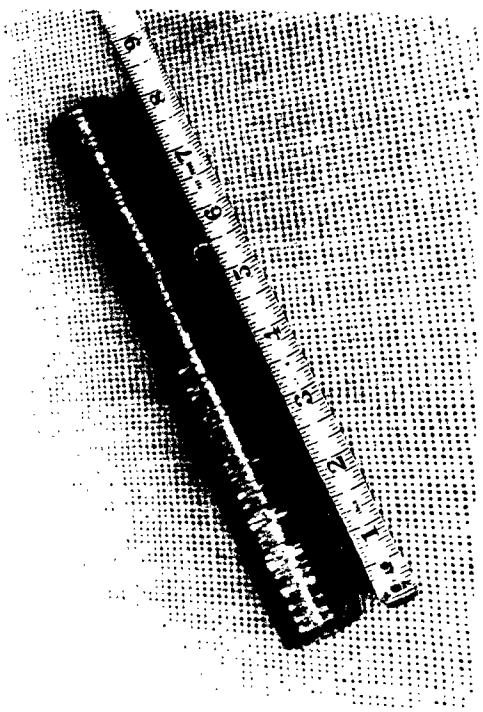


Fig. 2 - Large diameter unalloyed tungsten crystal DC [111].

Fig. 3 - Tungsten Rod - swaged 60% R.A.

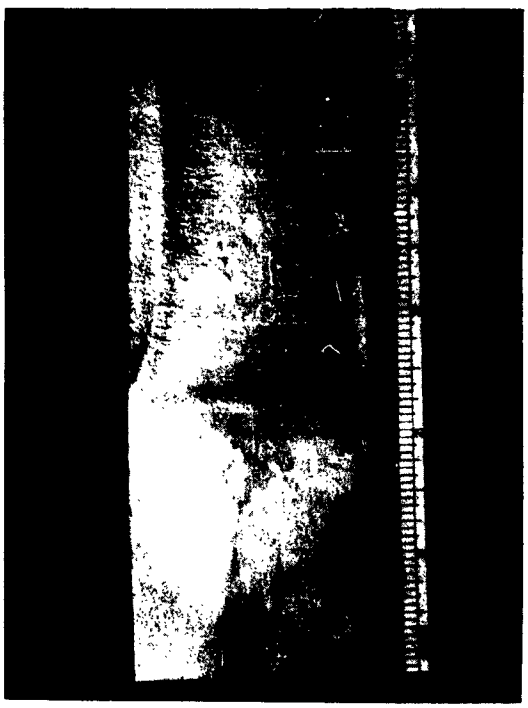


Fig. 4 - Tungsten Sheet - rolled to .040 inch.

6X

Fig. 5 - PCB [111], unalloyed tungsten crystal swaged to 60% R.A.

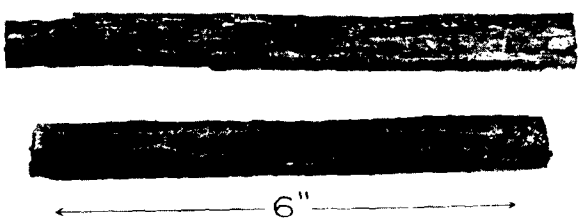


Fig. 6a - Tungsten alloys forged from .550 inch to .200 inch. RI contained 1% Ir and broke during forging. RG contained 1% Nb and represents the more normal case of successful forging.

6X

Fig. 6b - PGC [111], unalloyed tungsten crystal. This is the side view after being forged from .580 inch to .256 inch. Note the presence of deformation bands.

3X

Fig. 7 - RU-1 [111], 1% Nb alloy forged from .505 inch to .230 inch and rolled to .404 inch. Note the presence of deformation bands.

Fig. 8 - Transverse and longitudinal sections of PA, an unalloyed tungsten crystal with an [111] axis. The maximum misorientation was about 4-1/2°.

100X

Fig. 9a - PC [111], an unalloyed tungsten crystal showing the subgrain boundaries and etch pyramids.

750X

Fig. 9b - PC [111], an unalloyed tungsten crystal showing the triangular etch pyramids characteristic of the (111).

750X

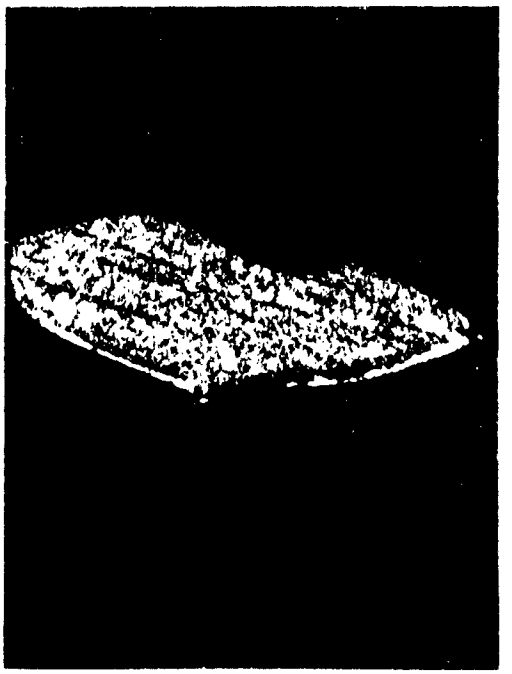
Fig. 10 - PA [111], an unalloyed tungsten crystal shown in Fig. 8. This is the longitudinal section showing the shingle-like structure characteristic of (h,k,O). Note the change in etching at the grain boundary.

3X

Fig. 11a - Seed end of large diameter unalloyed tungsten crystal DB [111]. Note the large number of subgrains and very slight difference in orientation.

3X

Fig. 11b - Cap end of large diameter unalloyed tungsten crystal DA [111]. Note the fewer (comparatively) subgrains and larger amounts of misorientation.



100X

Fig. 12 - PH [100], completely recrystallized showing shingle-like structure at the grain boundary, highly etched.

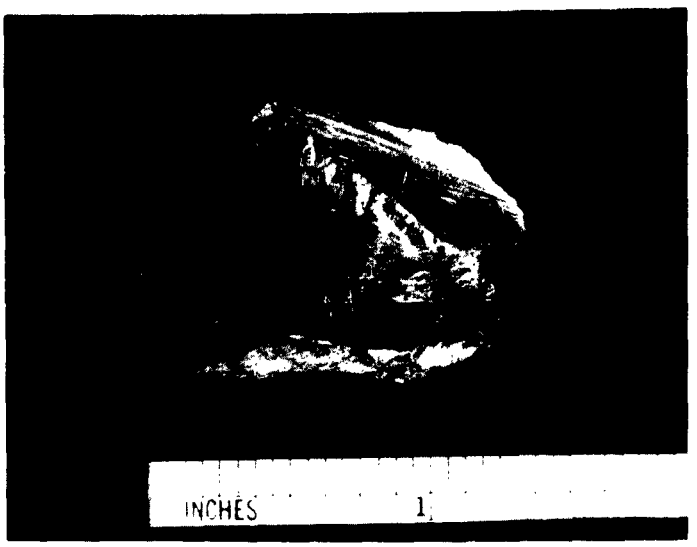


Fig. 13a - Photo of cleaved tungsten, the side to the upper right has been ground. Note the smooth planar effect.

10X

Fig. 13b - Photo of fracture along boundaries. Note the curved and uneven appearance.

FIGURE 14

HARDNESS VS % R.A. FOR SWAGED TUNGSTEN, ALLOYED & UNALLOYED

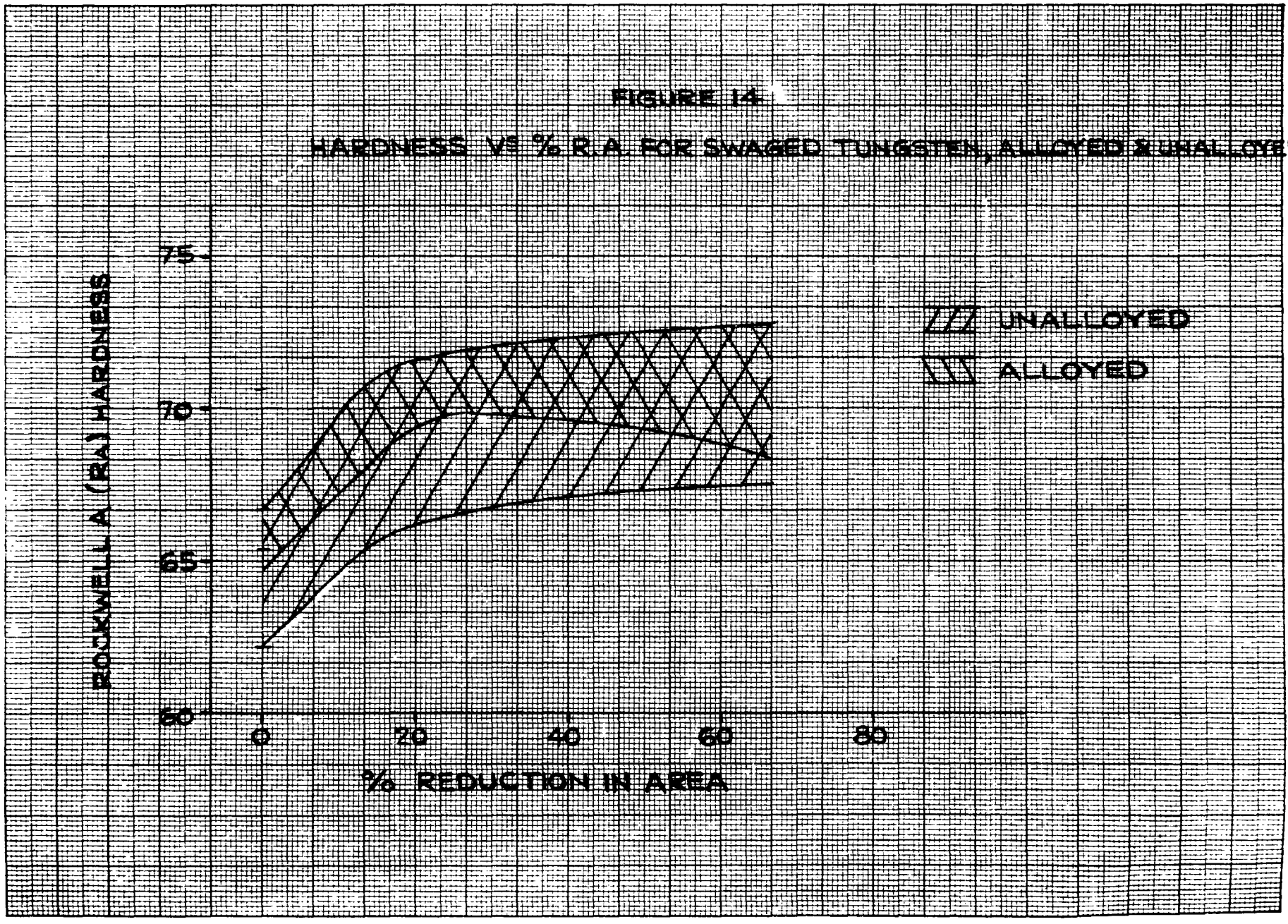


FIGURE 15a

ROOM TEMPERATURE HARDNESS VS TEMPERATURE, ½ HOUR ISOCHRONAL ANNEALS, FOR WORKED UNALLOYED TUNGSTEN (TYPICAL CRYSTALS)

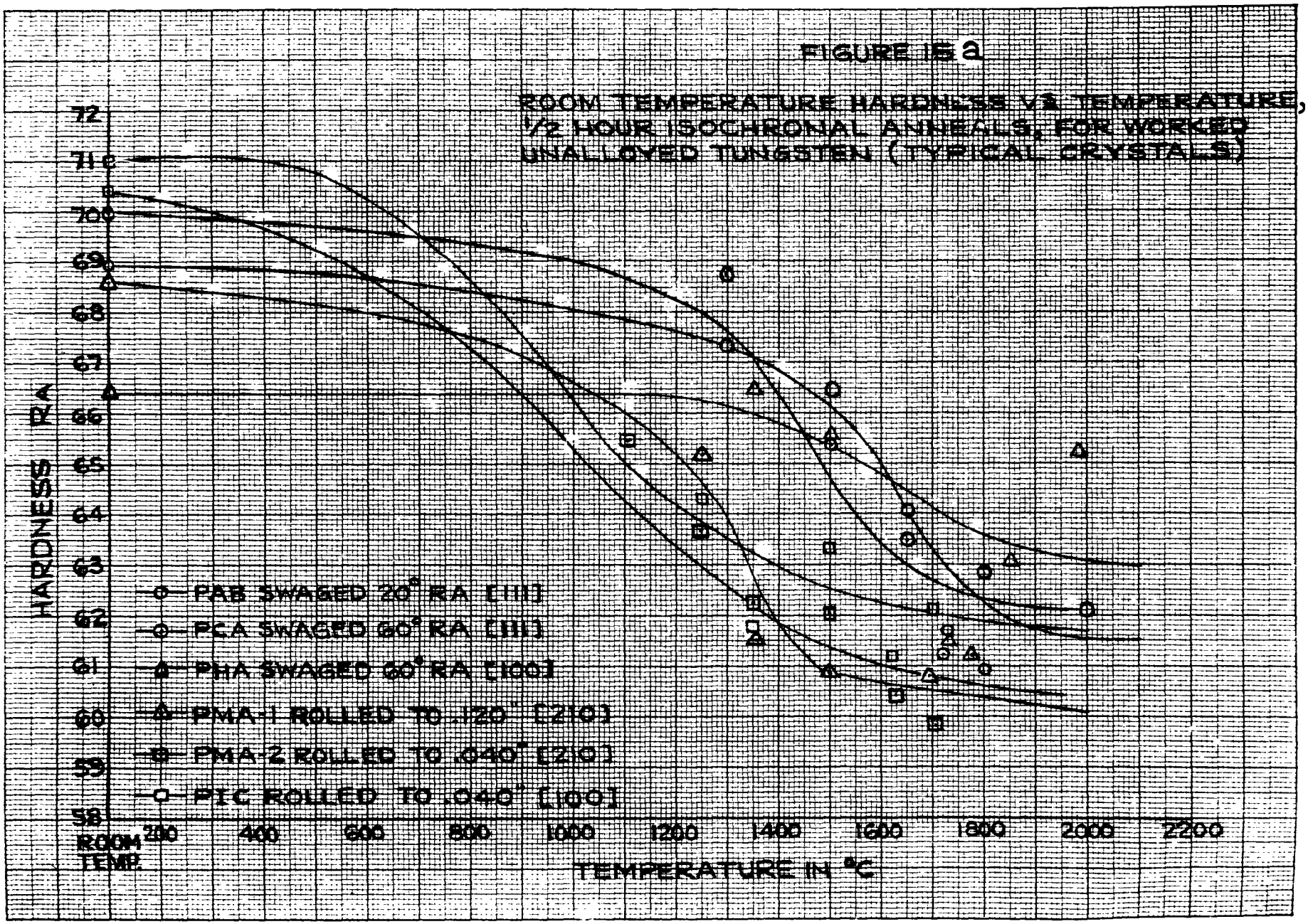


FIGURE 15½

ROOM TEMPERATURE HARDNESS VS TEMPERATURE, ½ HOUR ISOCHRONAL ANNEALS, FOR WORKED TUNGSTEN ALLOYS ALL [1][2]

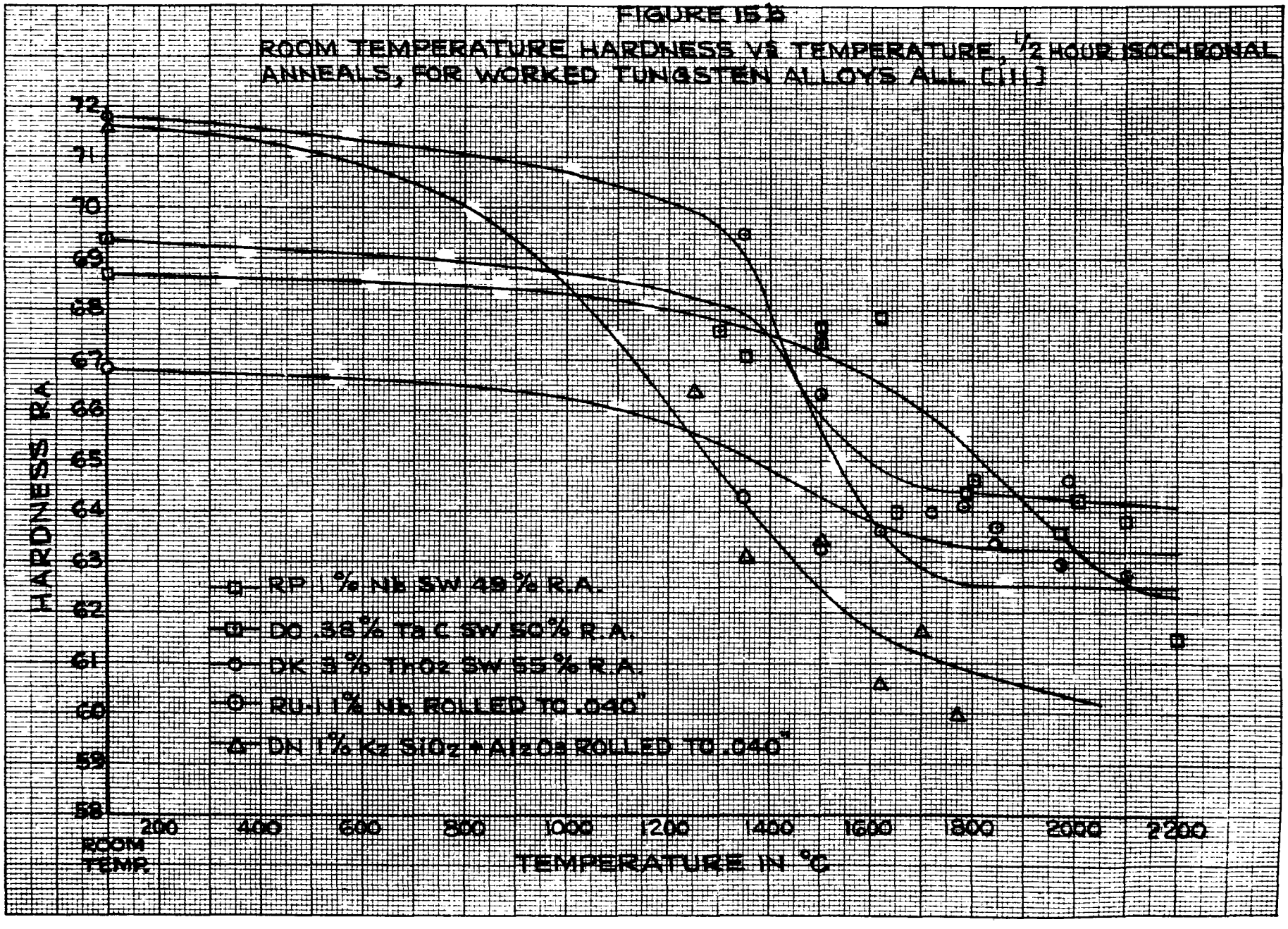


FIGURE 16A

ESTIMATED % OF MICROSTRUCTURE WHICH HAD RECRYSTALLIZED AS DETERMINED METALLOGRAPHICALLY VS TEMPERATURE FOR WORKED UNALLOYED TUNGSTEN (TYPICAL CRYSTALS)

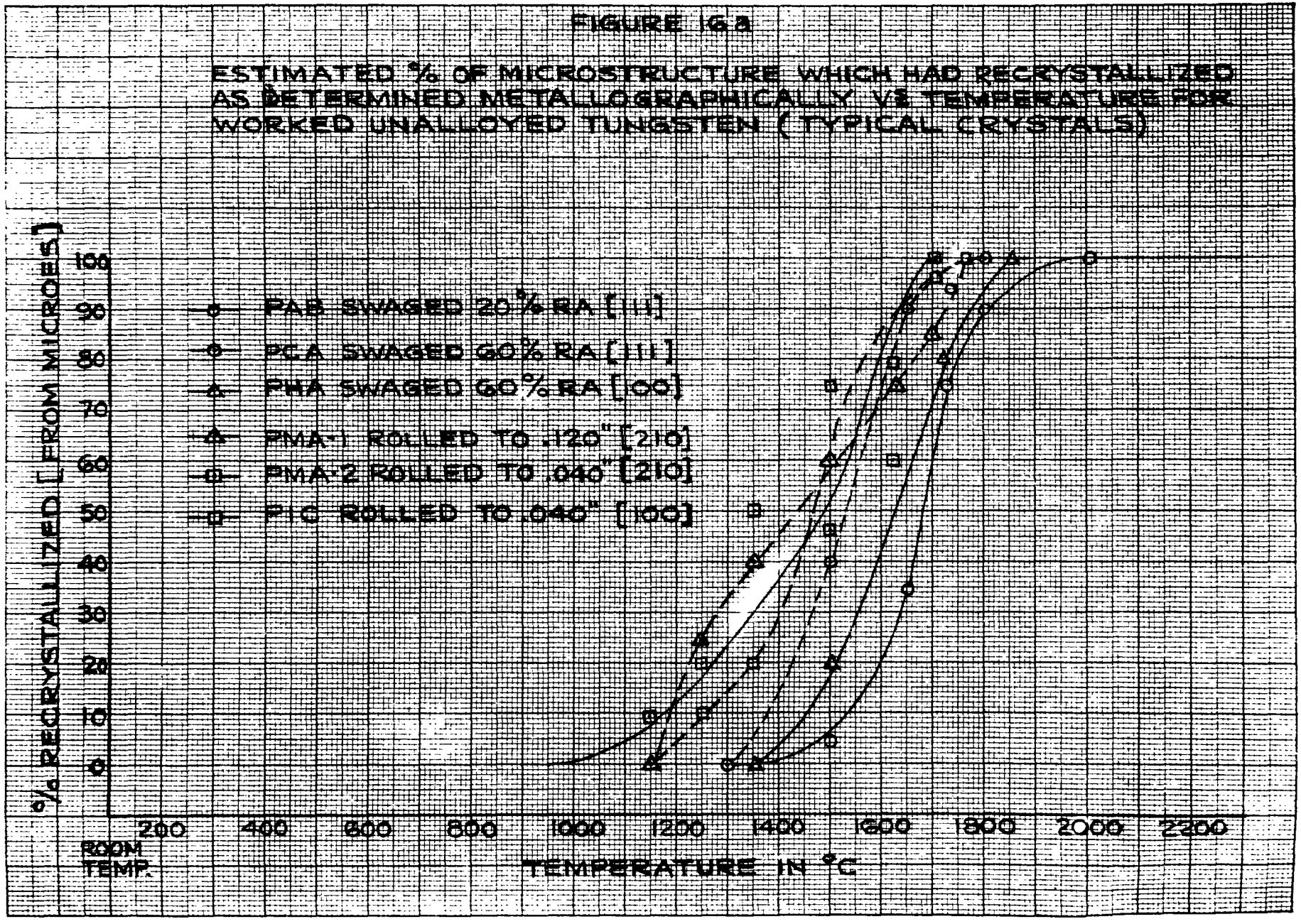
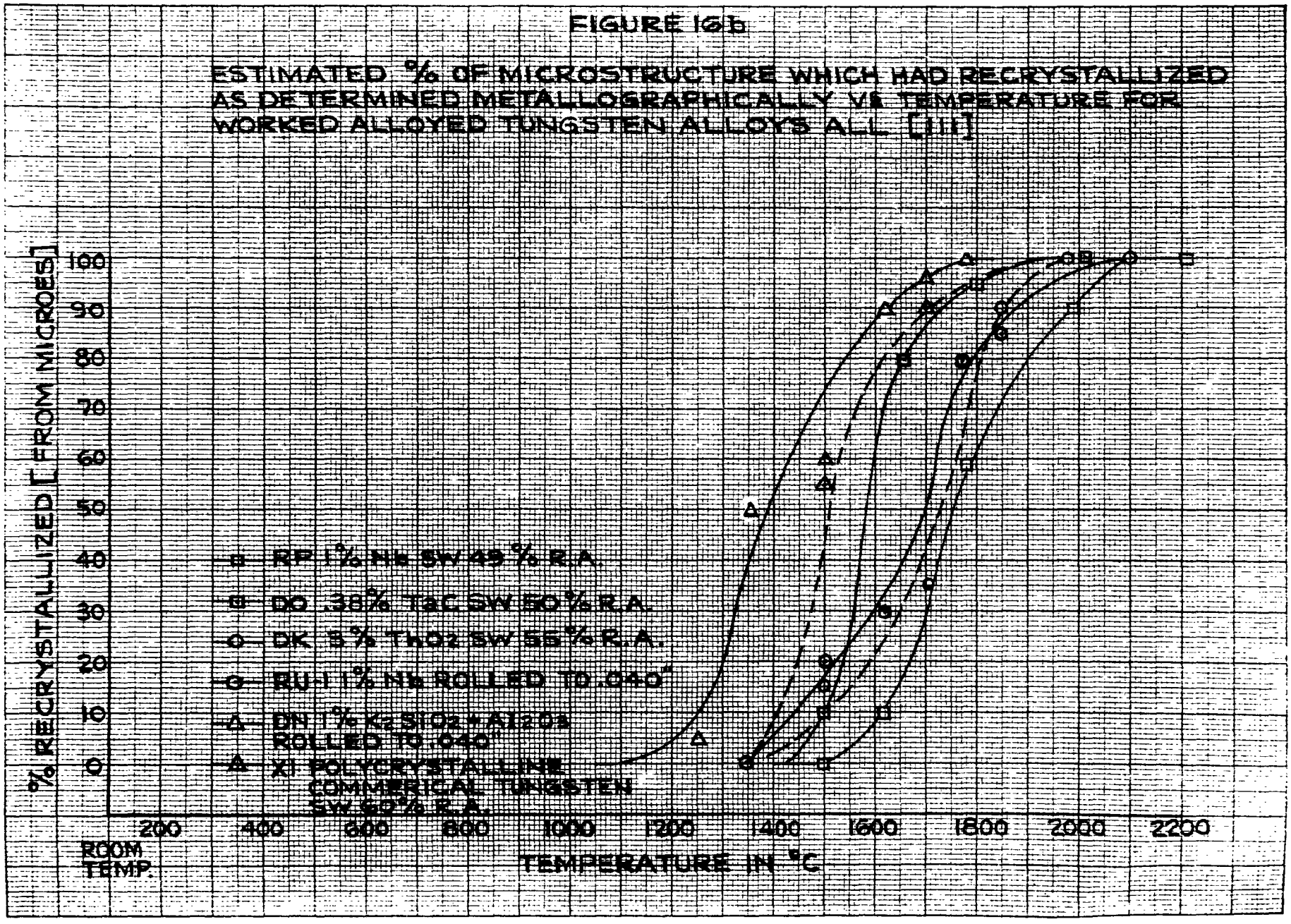
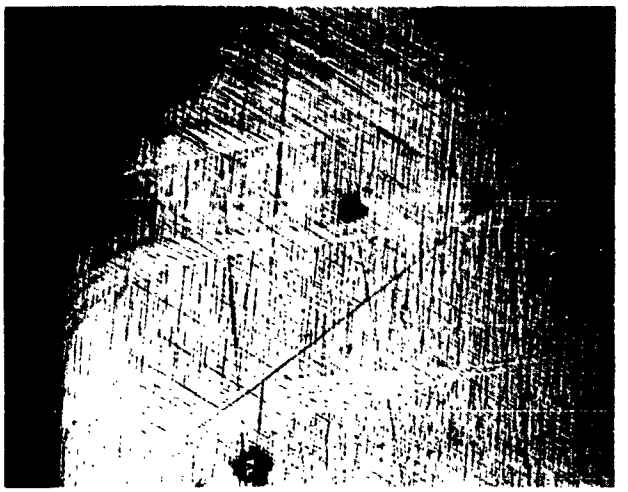


FIGURE 16b

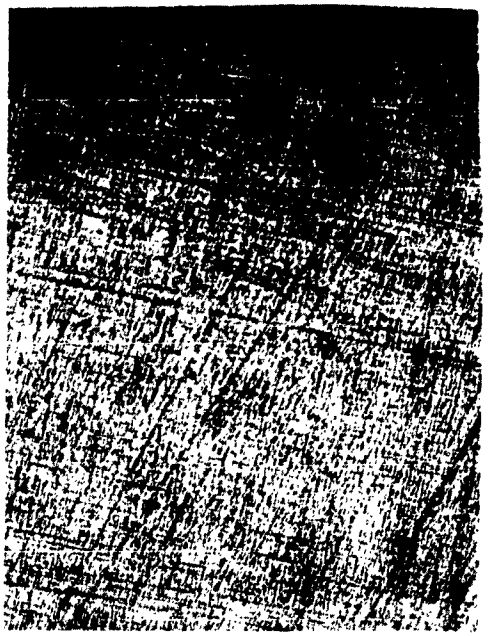
ESTIMATED % OF MICROSTRUCTURE WHICH HAD RECRYSTALLIZED AS DETERMINED METALLOGRAPHICALLY VS TEMPERATURE FOR WORKED ALLOYED TUNGSTEN ALLOYS ALL [111]





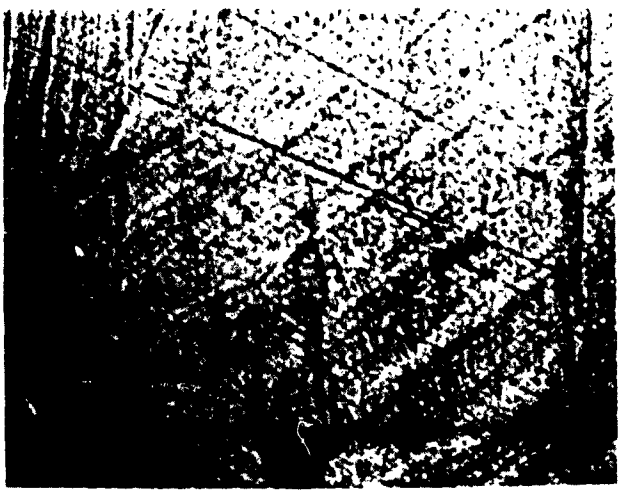
50X

Fig. 17a - PAB [111], a section of an unalloyed tungsten crystal swaged to 20% R.A. Note the presence of deformation bands.



50X

Fig. 17b - PAB, same as Fig. 17a, vacuum annealed at 1300°C for 1/2 hour.



50X

Fig. 17c - PAB, same as Fig. 17a, vacuum annealed at 1500°C for 1/2 hour.

500X

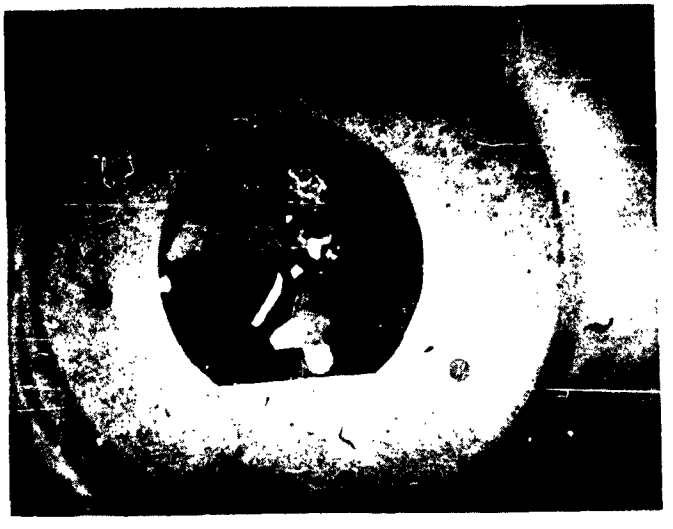
Fig. 17d - PAB, same as Fig. 17a, vacuum annealed at 1500°C for 1/2 hour. Note the start of recrystallization.

50X

Fig. 17e - PAB, same as Fig. 17a, vacuum annealed at 1650°C for 1/2 hour.

5(

Fig. 17f - PAB, same as Fig. 17a, vacuum annealed at 1725°C for 1/2 hour.

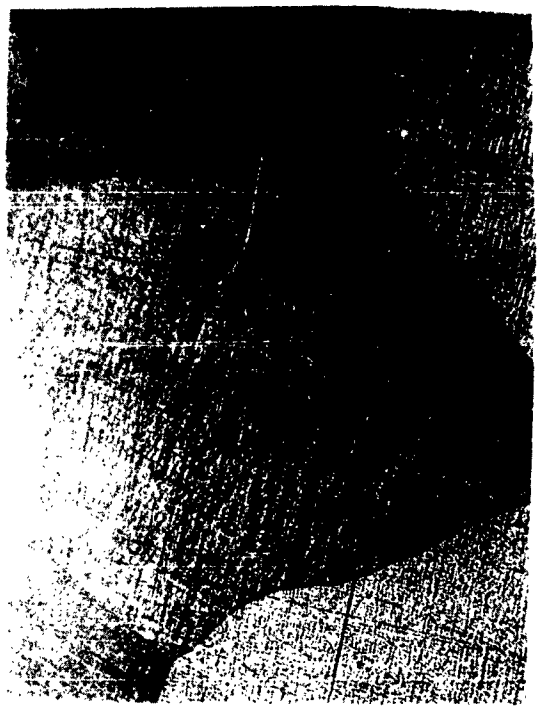


3X

Fig. 17g - PAB, same as Fig. 17a, vacuum annealed at 1725°C for 1/2 hour.

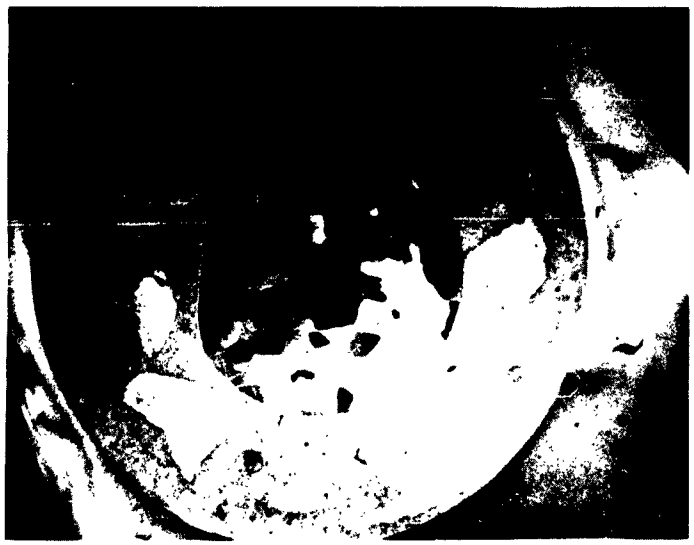
50X

Fig. 17h - PAB, same as Fig. 17a, vacuum annealed at 1800°C for 1/2 hour.



50X

Fig. 17i - PAB, same as Fig. 17a, vacuum annealed at 2000°C for 1/2 hour. Note recrystallization is complete.



3X

Fig. 17j - PAB, same as Fig. 17a, vacuum annealed at 2000°C for 1/2 hour.

150X

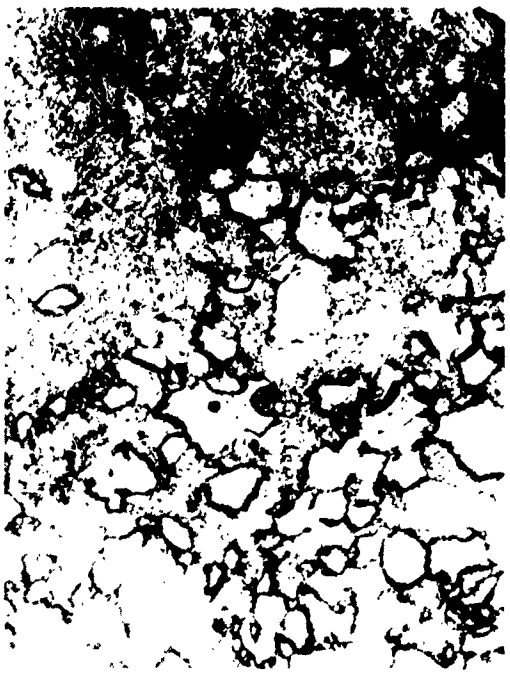
Fig. 18a - RU-1 [111], a section of an 1% Nb alloy forged from .505 inch to .230 inch and rolled to .040 inch. Refer to Fig. 7 which is at a lower magnification and one can see the presence of deformation bands.

150X

Fig. 18b - RU-1, same as Fig. 18a, vacuum annealed 1350°C for 1/2 hour.

500X

Fig. 18c - RU-1, same as Fig. 18a, vacuum annealed 1350°C for 1/2 hour.



150X

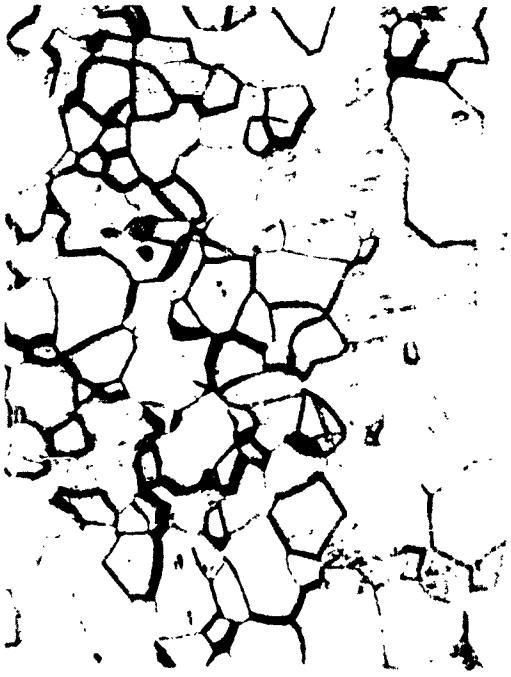
Fig. 18d - RU-1, same as Fig. 18a, vacuum annealed 1500°C for 1/2 hour.

150X

Fig. 18e - RU-1, same as 18a, vacuum annealed 1620°C for 1/2 hour.

150X

Fig. 18f - RU-1, same as Fig. 18a, vacuum annealed 1775°C for 1/2 hour. Very heavy etch.



150X

Fig. 18g - RU-1, same as Fig. 18a, vacuum annealed 1850°C for 1/2 hour.

150X

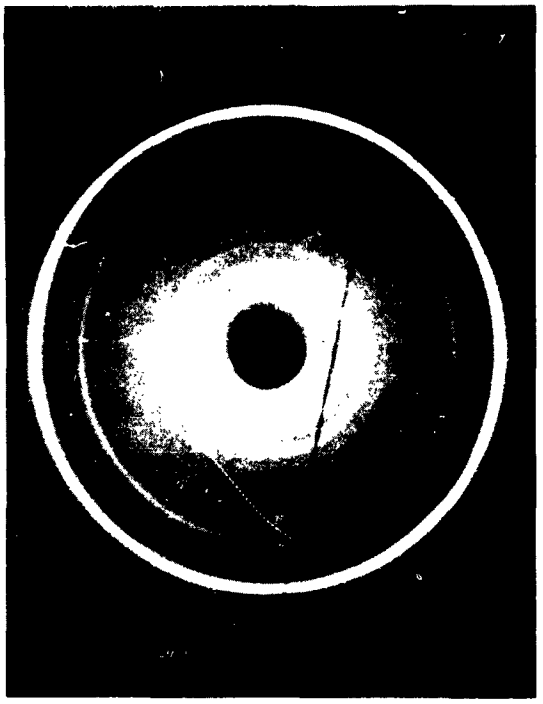
Fig. 18h - RU-1, same as Fig. 18a, vacuum annealed 1975°C for 1/2 hour.

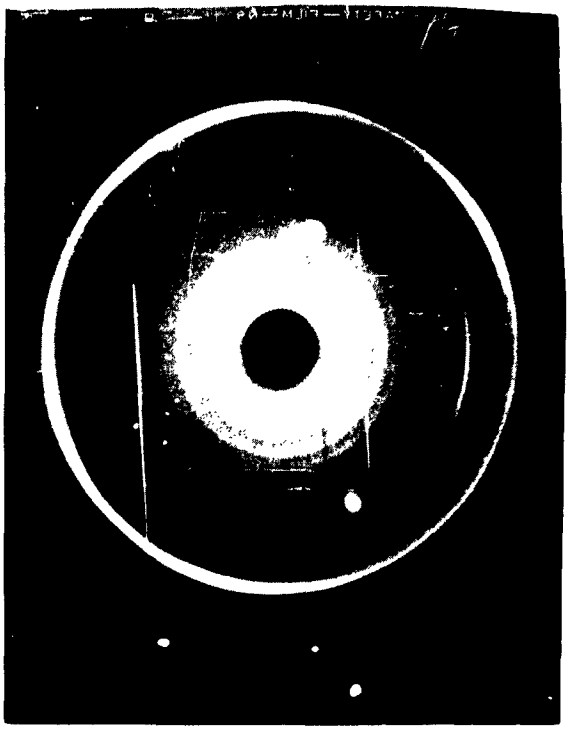
10X

Fig. 18i - RU-1, same as Fig. 18a, vacuum annealed 1975°C for 1/2 hour. Note that some coarsening has taken place. The unetched portion is where the electrical contact was made.

150X

Fig. 18j - RU-1, same as Fig. 18a, vacuum annealed at 2100°C for 1/2 hour. Note that recrystallization is complete.





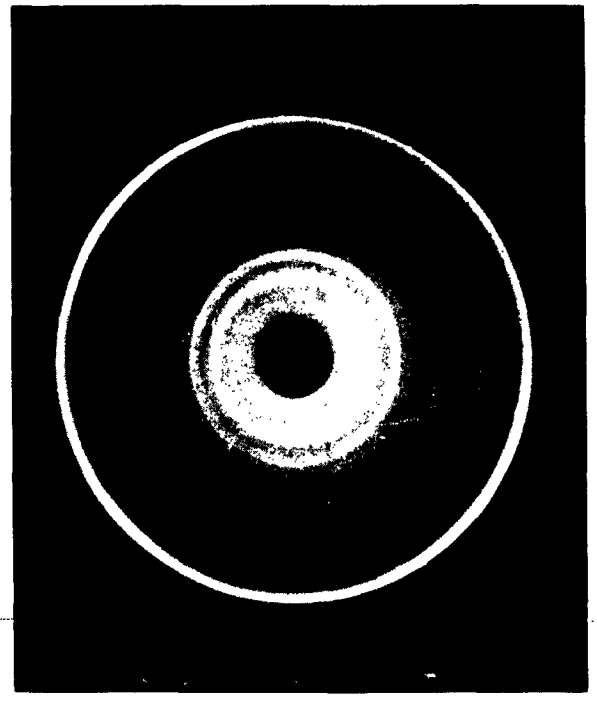


Fig. 19c - PJ [110]

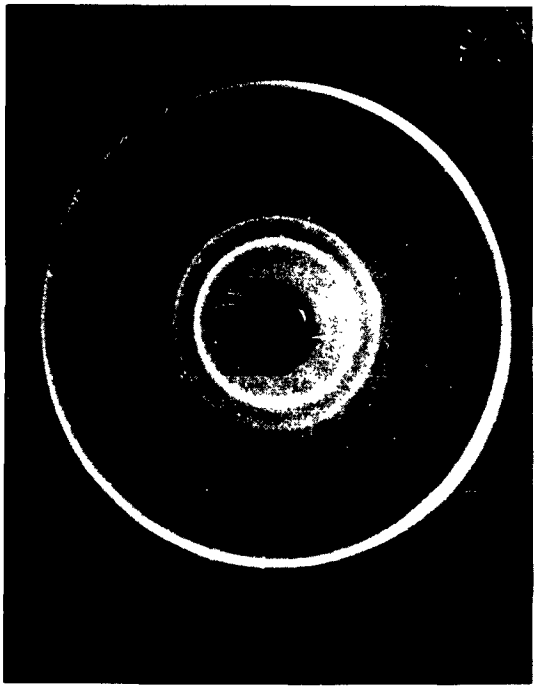


Fig. 19d - PM [210]

Figs. 19a through 19d - X-ray back-reflection photographs of tungsten sheet, 3 cm distance, rolling direction is vertical. Note the completeness and sharpness of the Debye rings.

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Fansteel Metallurgical Corp., 2200 Sheridan Road, North Chicago, Illinois

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